2.2.5 Limit test for arsenic

The limit test for arsenic is provided to demonstrate that the content of arsenic does not exceed the limit given in the individual monograph in terms of micrograms of arsenic per gram of the test substance.

To carry out the limit test for arsenic a solution is prepared from the test substance by a procedure specified in the monograph. This procedure assures that the solution in every case contains the whole of the arsenic (if any) present in the substance.

The standard stain against which the comparison is made contains 10 µg of As.

The procedure described may also be used to determine the amount of arsenic in the substance by matching the depth of colour of the stain with a series of standard stains. A stain equivalent to the 1 mL standard stain produced by operating on 10 g of a substance indicates that the amount of arsenic is 1 µg/g.

In the statements of arsenic limits, the permitted amount of arsenic is expressed as As.

Apparatus

A suitable type of apparatus is described below, though other acceptable constructions are available.

A wide-mouthed bottle of about 120 mL capacity, is fitted with a rubber bung through which passes a glass tube. The latter, made from ordinary glass tubing, has a total length of 200 mm and an internal diameter of exactly 6.5 mm (external diameter about 8 mm), is drawn out at one end to a diameter of about 1 mm, and has a hole not less than 2 mm in diameter blown in the side of the tube, near the constricted part. The tube is passed through the bung fitting the bottle so that, when inserted in the bottle containing 70 mL of liquid, the constricted end of the tube is above the surface of the liquid and the hole in the side is below the bottom of the bung. The upper end of the tube is cut off square, and is either slightly rounded off or ground smooth.

Two rubber bungs (about 25 mm × 25 mm), each with a hole bored centrally and true and exactly 6.5 mm in diameter, are fitted with a rubber band or spring clip for holding them tightly together. Alternatively, the two bungs may be replaced by any suitable construction satisfying the conditions of the test, as described below.

Recommended procedure

Pack the glass tube lightly with cotton-wool, previously moistened with lead acetate (80 g/l) TS and dried, so that the upper surface of the cotton-wool is not less than 25 mm below the top of the tube.

Insert the upper end of the tube into the narrow end of one of the pair of rubber bungs, either (1) to a depth of about 10 mm in the case of the tube with the rounded-off end or (2) so that the ground end of the tube is flush with the larger end of the bung. Place a piece of mercuric bromide paper AsR flat on the top of the bung, and place the other bung over it. Secure the assembly by means of a rubber band or spring clip, in such a manner that the borings of the two bungs (or the boring of the upper bung and the glass tube) meet to form a true tube 6.5 mm in diameter interrupted by a diaphragm of mercuric bromide paper AsR.

Instead of this method of attaching the mercuric bromide paper AsR, any other method may be used provided (1) that the whole of the evolved gas passes through the paper, (2) that the portion of the paper in contact with the gas is a circle 6.5 mm in diameter, and (3) that the paper is protected from sunlight during the test.

Place the solution, prepared as specified in the monograph, in the wide-mouthed bottle, add 1 g of potassium iodide AsR and 10 g of granulated zinc AsR, and place the prepared glass tube assembly quickly into position. Allow the reaction to proceed for 40 minutes. Compare any yellow stain that is produced on the mercuric bromide paper AsR, with a standard stain, produced in a similar manner with a known quantity of dilute arsenic AsTS. Make the comparison in daylight and immediately after simultaneous preparation of the test and standard stains; the stains fade on keeping.

The most suitable temperature for carrying out the test is generally about 40°C but, as the rate of evolution of the gas varies somewhat with different batches of granulated zinc AsR, the temperature may be adjusted to obtain a regular, but not too violent, evolution of gas. The reaction may be accelerated by placing the apparatus on a warm surface, care being taken to ensure that the mercuric bromide paper AsR remains quite dry throughout the test.

Between successive tests, the tube must be washed with hydrochloric acid (~250 g/l) AsTS, rinsed with water, and dried.

Standard stain

Prepare a solution by adding 10 mL of stannated hydrochloric acid (~250 g/l) AsTS and 1 mL of dilute arsenic AsTS, to 50 mL of water. The resulting solution, when treated as described in the general test, yields a stain on the mercuric bromide paper AsR, referred to as the standard stain.